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DOCKET NO: 214751US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF	:
KAZUTAMI MITANI, ET AL.	: EXAMINER: SALVATORE, LYNDA
SERIAL NO: 09/926,282	:
FILED: OCTOBER 5, 2001	: GROUP ART UNIT: 1771
FOR: PREFORM FOR COMPOSITE MATERIAL AND COMPOSITE MATERIAL	:

DECLARATION UNDER 37 C.F.R. § 1.132

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Now comes _____ who deposes and states that:

1. I am familiar with the invention claimed in the above-identified application.

2. I am a graduate of_____ university and received my _____ in the year ____.

3. I have been employed by _____ since _____ and I have been conducting research in the field of _____ for ____ years.

4. I have reviewed and understood the contents of the Ludwig (WO 92/06845) and Bompard (US 5,784,642) publications cited as prior art against the claims of the present application by the Examiner.

5. In order to compare the presently claimed invention with the disclosure of Ludwig and Bompard the following experiments were carried out by me or under my direct supervision and control.

6. Purpose

The present invention is compared with the citations. The present invention and the citations have "a layer which is made of thermoplastic resin and has space so that flow of liquid resin to be injected for molding composite material is uninhibited". The layer of the present invention has a weight of 1 to 50 g/m^2, and the layers of the citations have weights of less than 1 g/m^2 or more 50 g/m^2. In this experiment report, it is shown that the present invention has remarkable effects in mechanical properties in comparison with the citations.

7. Experiments and results

(1) Materials

(1.1) Thermoplastic resin threads

Nylon 12 manufactured by Daicel-Huls Ltd. was spun to produce thermoplastic resin threads having 67 dtex/36 filaments (0.0067 g/m).

(1.2) TP plying-wound layer

A peelable paper was provided on a drum of a drum winder manufactured by Fujita Machine Co., Ltd., and the thermoplastic resin threads were wound thereon with each pitch as described below so as to be 1, 10, 50, 100 g/m^2, respectively. Cellophane tape was provided

on the threads wound on peelable paper to prevent the pitch from changing, and then this laminate (containing the peelable paper) was opened by cutting to form a layer in which the thermoplastic resin threads were plying-wound by each pitch (hereinafter, the layer is called the TP layer).

Table 1

condition Weight per area (g/m^2)	Density (number/inch)	Pitch (mm)
0	-	-
1	3.8	6.7
10	37.9	0.67
50	189.6	0.134
100	379.1	0.067

(1.3) Reinforcing material formed of reinforcing fiber

As a reinforcing material formed of reinforcing fiber (hereinafter, referred to simply as the CF layer), "TRK979P" (carbon fiber uniaxial fabric, 200 g/m^2) manufactured by Mitsubishi Rayon Co., Ltd., was prepared.

"TRK979P" is made by weaving carbon fiber "TR50S 12L" manufactured by Mitsubishi Rayon Co., Ltd., as warp, and glass fiber "D450" (224 dtex/400 filament) manufactured by Unitika Glass Fiber Co., Ltd., as weft so as to have a weaving density of warp: 2.5 threads/cm and weft: 2 threads/cm.

(1.4) Liquid resin (matrix resin)

As a liquid resin, an ordinary temperature setting resin "DENATOOL" manufactured by Nagase ChemteX Corporation was prepared. The resin is made by mixing the following resin and hardener by the weight ratio of resin/hardener= 100/33.

Mixing ratio

Resin XNR6708

Bisphenol A type liquid epoxy resin 45% by weight,

Bisphenol F type liquid epoxy resin 20% to 30% by weight

Hardener XNH6708

Modified alicyclic polyamine (main content),

Diethylenetriamine 2% by weight

(2) Molding composite material

(2.1) Preform preparation

The TP plying-wound material (TP layer) and reinforcing material composed of reinforcement fiber (CF layer) were laminated according to the following laminating composition, and thereby a preform was prepared.

Table 2

Preform 1	(CF layer $0°)_8$	TP layer provided between CF layers
Preform 2	(CF layer +45°/CF layer 0°/CF layer -45°/CF layer $90°)_{2S}$	TP layer provided between CF layers
Preform 3	(CF layer $0°)_8$	-
Preform 4	(CF layer +45°/CF layer 0°/CF layer -45°/CF layer $90°)_{2S}$	-

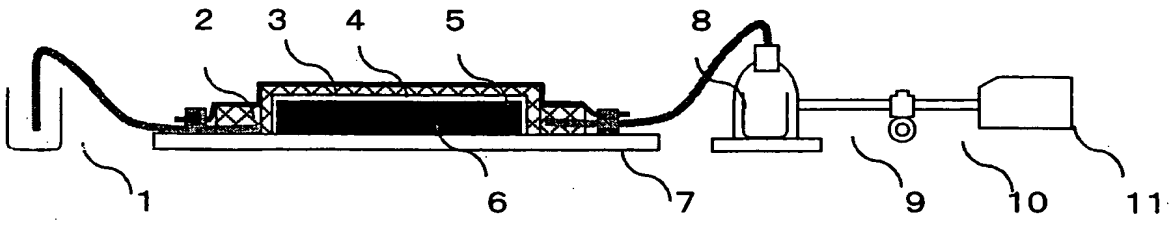
Subscripts indicate repetition number of each layer. Subscripted S means a mirror lamination.

(2.2) Molding

(a) Each preform was provided on a stainless steel plate, and a bag film was covered thereon (see the following figure).

(b) The liquid resin was injected while the inside of the bag film was decompressed, and maintaining injection and decompression until liquid resin contained no bubbles.

(c) The preform with the bag film was transferred into a furnace and was left at room temperature for 16 hours therein. The preform was heated to 60°C for two hours, and further heated to 120°C for 10 hours. The heated preform was cooled to room temperature, and then a composite material was obtained.



1: Resin
2: Sealant tape
3: Bag film
4: Infusion mesh
5: Nylon taffeta
6: Preform
7: Stainless steel plate
8: Nylon tube
9: Trap
10: Vacuum gage
11: Vacuum pump Evaluation

8. Evaluation

(3.1) Interlaminar shear strength (ILSS) test

With regard to a composite material made from each preform 1 and preform 3, ILSS test was conducted per five test pieces in accordance with ASTM D 2334-84, and using Instron's universal material tester 4465 manufactured by Instron Japan Co., Ltd.

(3.2) Bending test

With regard to a composite material made from each preform 1 and preform 3, bending test was conducted per five test pieces in accordance with ASTM D 790M, and using Instron's universal material tester 4465 manufactured by Instron Japan Co., Ltd.

(3.3) Compression after impact (CAI) test

With regard to a composite material made from each preform 2 and preform 4, in accordance with SACMA method SRM2-94 (impact energy: 360 inch-pound, n=2; this test was conducted per two test pieces), a damaged area was measured using ultrasonic detecting visualized system "SDS6500MR" manufactured by Krautkramer Japan Co., Ltd., and compression test was conducted using 50 t universal testing machine "UDH 50" manufactured by Shimadzu Corporation.

9. Evaluations and Results

Data of each composite material (using only $(0°)_8$) and evaluations and results are shown in Table 3.

Table 3

	Thermoplastic resin layer	g/cm^2	0	1	10	50	100
Composite material $(0°)_8$	Thickness	mm	1.76	1.73	1.87	2.29	3.06
	Comp density (ρ comp)	g/cm^3	1.55	1.54	1.50	1.42	1.33
	Carbon fiber content	% by vol.	58.3	57.6	51.0	39.1	25.5
Performance	ILSS (0°)	MPa	80.8	78.9	76.5	61.6	54.3
	ILSS (90°)	MPa	12.2	13.0	12.6	11.9	7.9
	FS (0°)	MPa	1397	1292	1389	974	649
	FS (90°)	MPa	69	79	86	65	48
	FM (0°)	GPa	87.4	91.7	84.7	72.7	52.4
	FM (90°)	GPa	5.7	6.1	5.7	4.9	4.0
	CAI damaged area (360in-lb)	cm^2	21.7	19.0	16.0	9.5	11.2
	comparing with 0g/m^2	%	100	87.6	73.5	43.8	51.6
	CAI strength (360in-lb)	MPa	147	156	153	157	137

Fig. 1

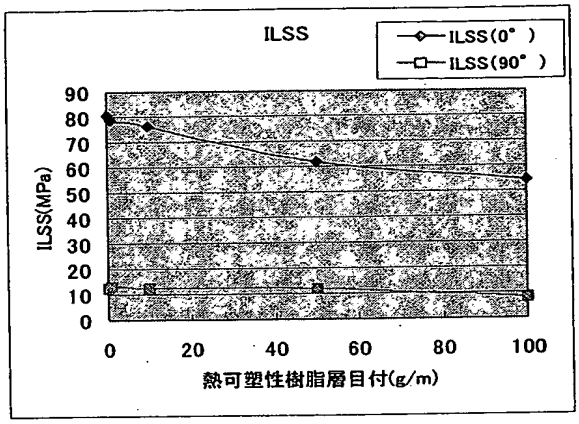


Fig. 2

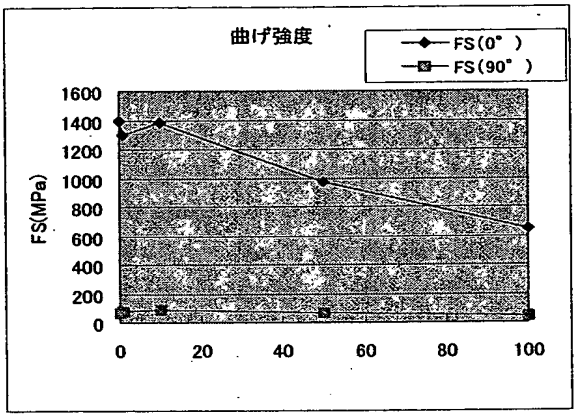


Fig. 3

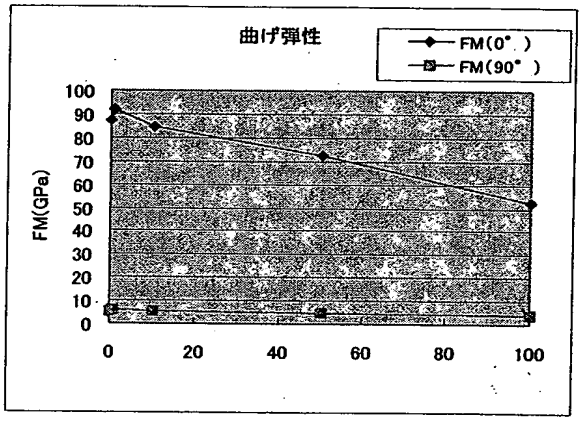


Fig. 4

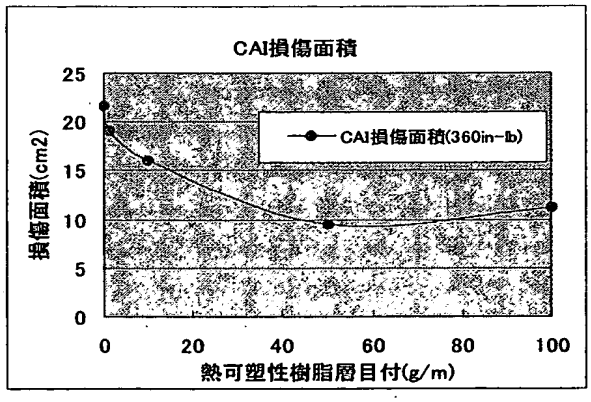


Fig. 5

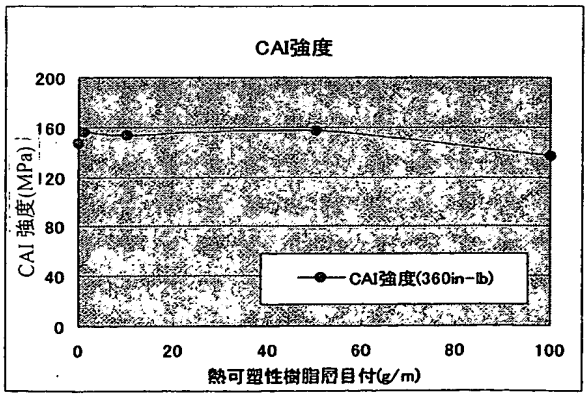


Fig. 1 ILSS; ISLL (MPa); Thermoplastic resin layer (g/m)

Fig. 2 Bending strength; FS (MPa)

Fig. 3 Bending elasticity; FM (GPa)

Fig. 4 CAI damaged area; Damaged area (cm^2); Thermoplastic resin layer (g/m); -•- CAI damaged area (360in-lb)

Fig. 5 CAI strength; CAI strength (MPa); Thermoplastic resin layer (g/m); -•- CAI damaged area (360in-lb)

10. Conclusion

(a) Damaged area can be reduced by inserting a TP layer; however, the effect is not improved even if the TP layer has more than $50g/m^2$.

(b) CAI can be improved by inserting a TP layer; however, the effect is not improved even if the TP layer has more than $50g/m^2$.

(c) On the other hand, ILSS and Bending strength 0° can be reduced by inserting a TP layer; however, the reduction cannot be considered to be significant since the damaged area is reduced and CAI is improved until the TP layer has more than $50g/m^2$.

11. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

12. Further deponent saith not.

_____
Signature

_____
Date

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IN RE APPLICATION OF :

KAZUTAMI MITANI, ET AL. : EXAMINER: SALVATORE, LYNDA

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FILED: OCTOBER 5, 2001 : GROUP ART UNIT: 1771

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DECLARATION UNDER 37 C.F.R. § 1.132

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Now comes Shigetsugu Hayashi who deposes and states that:

1. I am familiar with the invention claimed in the above-identified application.

2. I am a graduate of Osaka university and received my Master's Degree in the year 1984.

3. I have been employed by Mitsubishi Rayon Co., Ltd. since 1984 and I have been conducting research in the field of Advanced Composites materials for 17 years.

4. I have reviewed and understood the contents of the Ludwig (WO 92/06845) and Bompard (US 5,784,642) publications cited as prior art against the claims of the present application by the Examiner.

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Subscripts indicate repetition number of each layer. Subscripted S means a mirror lamination.

(2.2) Molding

(a) Each preform was provided on a stainless steel plate, and a bag film was covered thereon (see the following figure).

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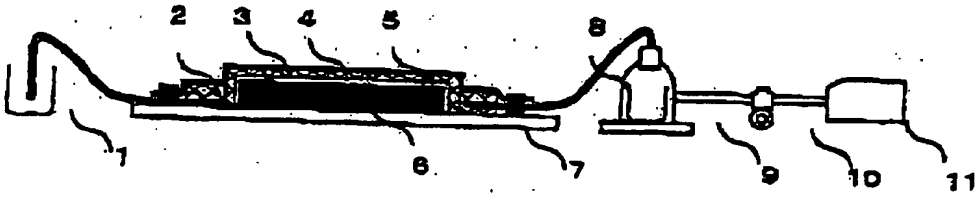

1: Resin
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7: Stainless steel plate
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8. Evaluation

(3.1) Interlaminar shear strength (ILSS) test

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With regard to a composite material made from each preform 1 and preform 3, bending test was conducted per five test pieces in accordance with ASTM D 790M, and using Instron's universal material tester 4465 manufactured by Instron Japan Co., Ltd.

(3.3) Compression after impact (CAI) test

With regard to a composite material made from each preform 2 and preform 4, in accordance with SACMA method SRM2-94 (impact energy: 360 inch-pound, n=2; this test was conducted per two test pieces), a damaged area was measured using ultrasonic detecting visualized system "SDS6500MR" manufactured by Krautkramer Japan Co., Ltd., and compression test was conducted using 50 t universal testing machine "UDH 50" manufactured by Shimadzu Corporation.

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	ILSS (90°)	MPa	12.2	13.0	12.6	11.9	7.9
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	FS (90°)	MPa	69	79	86	65	48
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Fig. 1

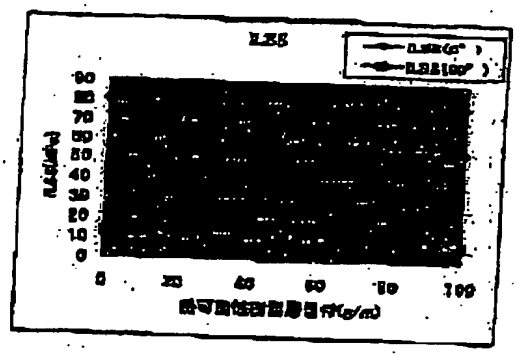

Fig. 2

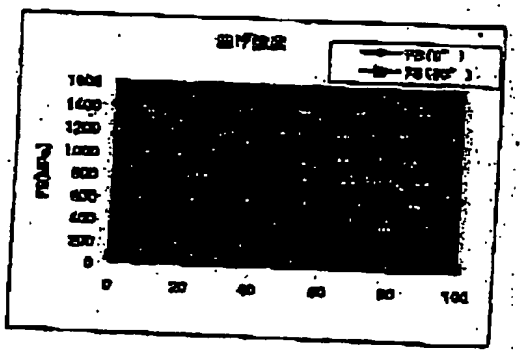

Fig. 3

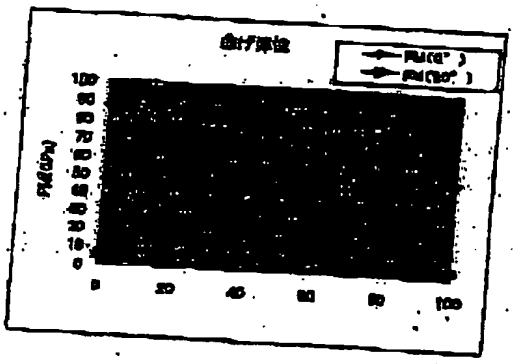

Fig. 4

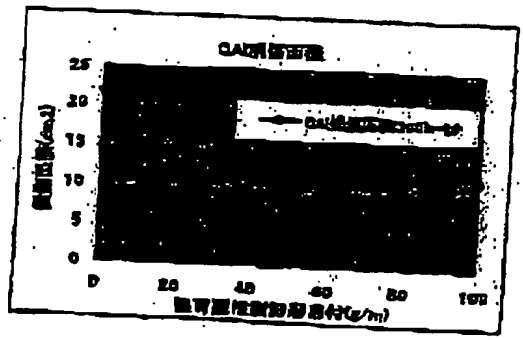

Fig. 5

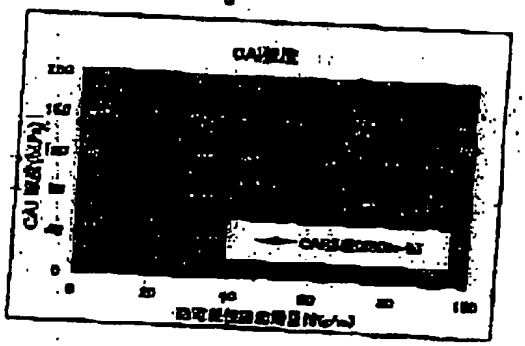

Fig. 1 ILSS; ISLL (MPa); Thermoplastic resin layer (g/m)

Fig. 2 Bending strength; FS (MPa)

Fig. 3 Bending elasticity; FM (GPa)

Fig. 4 CAI damaged area; Damaged area (cm^2); Thermoplastic resin layer (g/m); -▲- CAI damaged area (360in-lb)

Fig. 5 CAI strength; CAI strength (MPa); Thermoplastic resin layer (g/m); -▲- CAI damaged area (360in-lb)

10. Conclusion

(a) Damaged area can be reduced by inserting a TP layer; however, the effect is not improved even if the TP layer has more than 50g/m^2.

(b) CAI can be improved by inserting a TP layer; however, the effect is not improved even if the TP layer has more than 50g/m^2.

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Declaration Under 37 C.F.R. § 1.132

12. Further deponent saith not.

Signature

Jan. 09, 2004
Date